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Report No. 10

MONTHLY SCIENTIFIC PROGRESS REPORT

For the month of April 1961

STRESS CORROSION OF HIGH STRENGTH STEELS AND ALLOYS; ARTIFICIAL ENVIRONMENT

Research Project No, 389-1

Sponsored by
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ABSTRACT

Cumulative U-bend and bent beam stress corrosion test results on selected high strength missile steels and alloys are presented. The materials under test include AM355, PH15-7 Mo, B120 VCA, Vascojet 1000, 300M, D6Ac, 4137 Co, Rocoloy 270, and Ardeform 301.

A heat treating survey of the Vascojet primary heat is presented. The information will be used in heat treating sample material to the various stress levels for stress corrosion testing.

I. INTRODUCTION

The project work described herein represents a portion of a grant made available by the Army to promote a general scientific advancement in the area of case materials for missiles. This specific project is concerned with the synthetic environment stress corrosion testing of specified high strength steels and alloys. The research objectives of the project were presented in the July Monthly Scientific Report, Report No. 1.

Natural environmental tests on high strength steels and alloys are being conducted by Aerojet General Corporation, with actual production environments and rocket propellant environments being utilized. By prior mutual agreement, the same steel and alloy sheet material will be used for both projects and possible heat treatment variations will be circumvented by exchanging heat treated material whenever possible.

A number of drawings and schematic diagrams of apparatus and test methods pertinent to the project have been presented in prior reports. In addition, summarized surveys of applicable industrial and military literature have also been presented previously (August and September reports).

This report presents further information on continuing U-bend and bent beam tests for the assigned alloys, and for Rocoloy 270 and 4137 Co missile steels.

II. EXPERIMENTAL PROCEDURES

Test Methods

A discussion of the U-bend and bent beam test methods together with an outline of the synthetic stress corrosion test environments used in performing the research are given in the July, 1960, Monthly Scientific Report, Report No. 1.

All recently-exposed (and future) samples being subjected to stress corrosion testing are being weighed prior to exposure to the test solutions to provide, whenever possible, general corrosion information.

Apparatus

Schematic drawings of bent beam sample holders, the U-bend test and holders, a sample bending device for bent beam specimens, and a stress corrosion test tank were presented in Report Nos. 1 and 3.

Polyethylene containers are presently being used for the stress corrosion exposure of U-bend specimens to the various synthetic environments. Each container will adequately hold six U-bend specimens. The use of these containers will supplement the samples presently being tested in epoxy-coated tanks.

Construction of shelving and an aeration system (Report No. 6) together with other pertinent items has facilitated (wherever feasible) the transfer of samples under test from the glass containers to the epoxy-coated test tanks. The completed facility is illustrated in Report No. 7.

Alloy Sample Material

The selected alloys representative of the six groups to be tested include:

1. Low Alloy: Ladish D6AC.
2. Si-Modified 4300 Series: 300 M.
3. Hot-Worked Die Steel: Vascojet 1000.
4. Cold-Worked PH Steel: AM355.
5. Heat-Treated PH Steel: PH15-7 Mo.
6. Titanium Alloy: B120 VCA.

The cold-worked austenitic steel group was previously omitted because high strength steels of this type were not being used in any appreciable quantity for rocket motor cases or any similar type vehicle. However, by mutual agreement with the contract supervisor, a sample of 25% nickel alloy will be tested along with the other alloys when the material becomes available.

Aerojet General Corporation, on a cooperative stress corrosion program, has procured the first three of the foregoing alloys and heat treatment surveys are reportedly complete. All of the latter three alloys (the AM355, B120 VCA, and PH15-7 Mo) have been received by this Project, one-half of each having been forwarded to Aerojet.

A number of AM355 Primary Heat, Secondary Direction (highest Y.S., or rolling direction), bent beam samples have recently been exposed to stress corrosion testing. Additional U-bend samples of this material are being wet-ground to size.

The B120 VCA material will be subjected to testing upon completion of the heat treatment survey. A number of samples of as-received material are presently undergoing stress corrosion testing.

Comparative heats of D6Ac, 300M, Vascojet 1000, B120 VCA, PH15-7 Mo, and AM355 are on order or have been received. This material is intended for use as a check on the Primary Heat material undergoing stress corrosion testing (to determine whether or not the Primary Heat is a representative sample). The Rocoloy 270, however, is a low alloy, high strength steel similar to 4137 Co.

The physical properties of all three comparative heats of Vascojet 1000 were presented in Report No. 7 (page 7). The physical properties of the Primary Heat of Vascojet 1000 and the Primary Heat of B120 VCA, in the "as received" condition, are given in Table I.

The heat treating survey on the Primary Heat of Vascojet 1000 has been completed and is shown in Table II and Figure I. The following procedure was used in making this survey: all specimens (tensile and metallographic) were preheated to 1450°F, held for 30 minutes at temperature, austenitized at 1900°F for 40 minutes and air quenched.

TABLE I

PHYSICAL PROPERTIES OF VASCOJET 1000 AND B120 VCA

Type of Alloy	Yield Str., Kpsi (.2% Offset)	Tensile Str., Kpsi	Fracture Str., Kpsi	Red. in Area, %	Elongation in 1"	Elongation in 2"	Rolling Direction
Vascojet 1000 Primary Heat	75	103	174	51.1	26.7	19.0	Primary
	91	114	187	49.4	27.3	18.8	Secondary
B120 VCA Primary Heat	139	140	195	36.4	28.5	19.5	Primary
	143	145	192	35.9	29.0	19.5	Secondary

Note: Average of 3 specimens per direction.
Nominal specimen thickness: Vascojet 1000 (.070") and B120 VCA (.035").
All specimens in the "as received" condition.

TABLE II

PHYSICAL PROPERTIES OF VASCOJET 1000 (PRIMARY HEAT)

Tempering Temperature °F	Yield Str., Kpsi (.2% Offset)	Tensile Str., Kpsi	Fracture Str., Kpsi	Hardness R_c	Elongation in 1"	Elongation in 2"	Y.S./T.S.	Red. in Area, %
800	213	309	338	55.3	12.0	9.0	.689	14.4
850	220	318	338	56.0	--	--	.692	--
900	217	314	343	55.8	6.0	3.5	.691	14.0
950	228	320	348	56.0	7.0	4.0	.712	10.4
975	243	313	336	55.2	11.0	7.5	.776	13.0
1000	236	304	328	54.6	11.0	7.2	.776	18.3
1050	232	279	296	51.6	10.0	6.2	.831	18.3
1100	200	240	282	47.3	12.3	7.7	.833	26.2

Note: All results are the average of 3 specimens.
Nominal specimen thickness: .070".
Specimens taken in the secondary (highest Y.S.) direction.

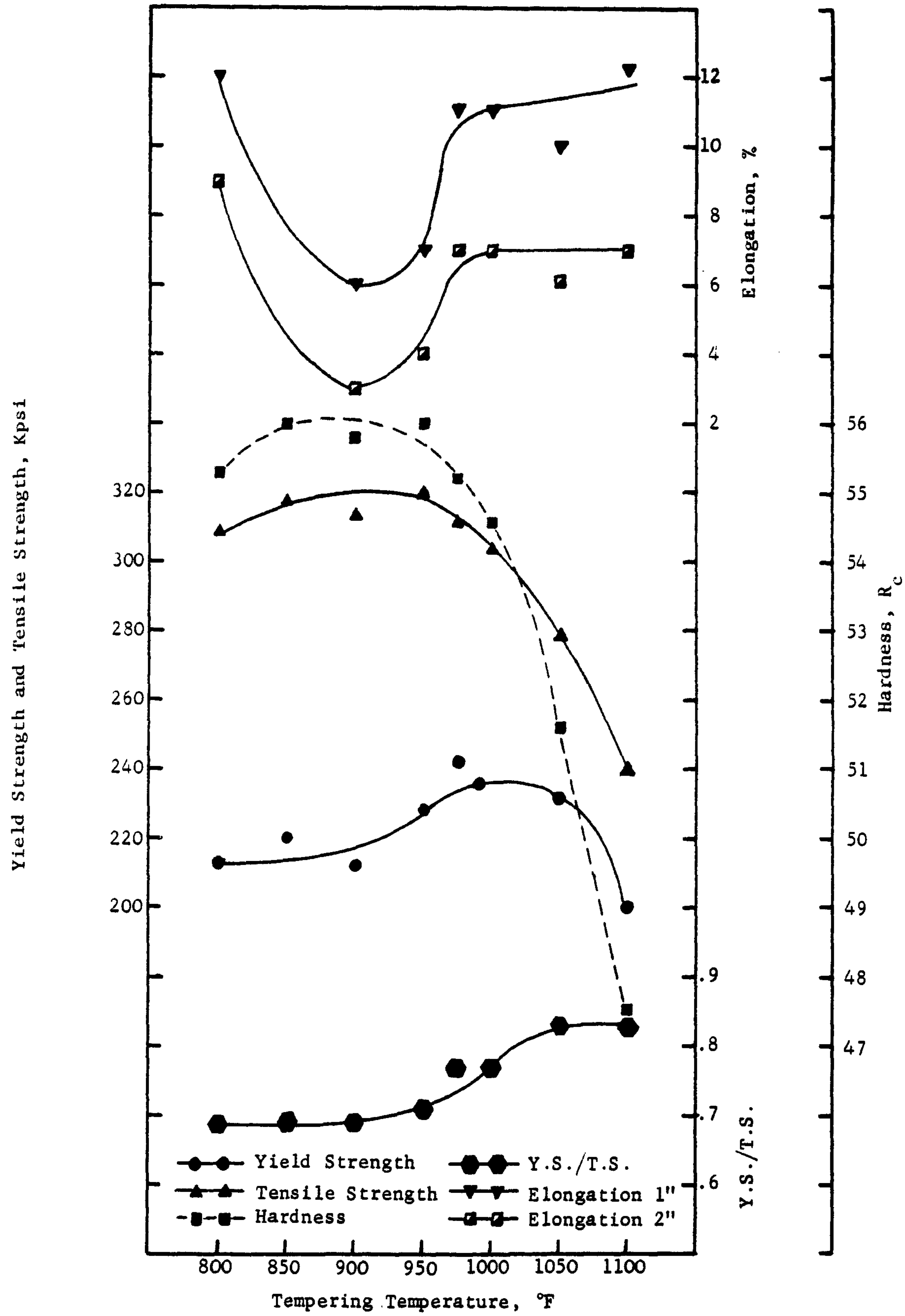

FIG. 1 - VASCOJET HEAT TREATMENT SURVEY

One metallographic specimen was removed after quenching. The specimens were then given a triple temper of two hour duraction (2 + 2 + 2 hrs.) between the levels of 800°F and 1100°F at 50°F increments, with an intermediate point of 975°F being included. All tensile specimens were heat treated in triplicate at each tempering temperature. Metallographic specimens were periodically removed during the tempering cycle.

After the first temper of two hours, metallographic specimens were removed from the 800°F and 1100°F specimens; these being the two extremes of the tempering survey. Another set of metallographic specimens were removed after the second two-hour temper at all tempering temperatures (800°F thru 1100°F). The final set of metallographic specimens were taken after the third two-hour temper, along with the tensile specimens. After each temper the specimens were air quenched. Hardness readings were recorded on all metallographic specimens taken during the tempering cycle.

Sample material of a stretch-formed austenitic stainless steel, referred to as Ardeform 301, has been supplied to this project by Arde-Portland, Inc. Two pieces of Ardeform 301 were received and will be called Sample No. 1 and Sample No. 2.

Sample No. 1 was cut (by sawing, wet cut-off wheel, wet grinding, drilling, and hand polishing) into "longitudinal" and "transverse" U-bend specimens as illustrated in Figure 2. The curved geometry of the samples

made the specimen preparation somewhat more difficult than was anticipated. However, specimen preparation was carefully undertaken to minimize induced stress in the specimens.

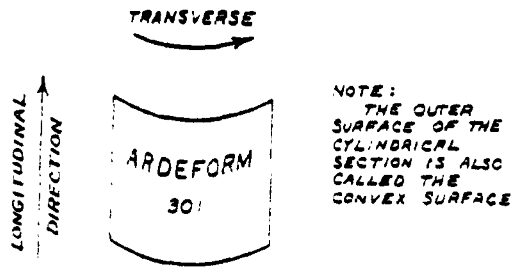

FIG. 2 - ASSUMED DIRECTIONS FOR ARDEFORM 301 SAMPLE MATERIAL

Forty "longitudinal" U-bend specimens and 30 transverse specimens were cut from Sample No. 1. Two "longitudinal" tensile specimens were taken from Sample No. 1. Sample No. 2 is being prepared in a similar manner. Table III shows the physical and chemical properties of Sample No. 1 and Sample No. 2.*

The 40 longitudinal specimens (Sample No. 1) were divided into 5 groups of 8 specimens. Each group of specimens has been bent, clamped, and immersed in the 5 test solutions being used by this project (Report No. 1, page 6).

Of the 8 specimens per longitudinal group, 4 were bent with the outer surface as the outside surface (tensile) of the U-bend and 4 with the inner surface as the outside tensile surface of the U-bend. For simplicity we have been calling these specimens "outside-out" and "inside-out", respectively, for want of a better descriptive reference.

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* Chemical properties supplied by Arde-Portland, Inc.

TABLE III

PHYSICAL AND CHEMICAL PROPERTIES OF ARDEFORM 301 SAMPLES

	Yield Str., Kpsi (.2% Offset)	Tensile Str., Kpsi	Fracture Str., Kpsi	Red. in Area, %	Elongation in 1"	Elongation in 2"	Test Direction
			Physical Properties				
Sample No. 1	183	229	293	29.0	10.5	7.7	Longitud.
Sample No. 2	209	235	295	29.2	9.0	6.0	Longitud.

Chemical Analysis

	C	Mn	P	S	Si	Cr	Ni
Sample No. 1	.055	1.52	.030	.016	.49	17.02	7.5

Sample No. 1: (Stretch-formed at -320°F)

Sample No. 2: Identical analysis
(Stretch-formed at -320°F followed by heat treatment at 500°F for 20 hrs.)

The 30 transverse specimens were divided into 5 groups, 6 specimens to a group. The curvature of the specimens prevented exposure of both inside and outside specimen surfaces as the test surface in tension of the U-bend. Consequently all specimens in the transverse direction are "outside-out."

All specimens were finished with 240-grit paper as a final step in preparation before bending. Degreasing after bending was accomplished with acetone followed by chloroform, thus eliminating surface film residue as a factor.

Sample No. 2 has been cut into specimens and will be in the test solutions in the near future.

III. EXPERIMENTAL

As the previous section of this report shows, sample preparation is continuing on the AM355, B120 VCA, PH15-7 Mo and the various comparative heats of all six assigned alloys.

U-bend and bent beam stress corrosion testing for the high strength, low alloy 4137 Co missile steel and U-bend tests for the low yield strength level (135 Kpsi, as received) B120 VCA are continuing. A number of AM355 bent beam samples have also been cut to length and exposed to stress corrosion environments in the laboratory. U-bend samples of AM355, Ardeform 301, and PH15-7 Mo have been prepared and immersed in test solutions. In addition, bent beam samples of 4137 Co have been mounted on an outdoor test rack for heavy industrial environment exposure.

A more detailed description of the various tests in progress includes:

Bent Beam Tests

Cooperative bent beam stress corrosion tests are being conducted with 4137 Co missile steel samples by the U. S. Steel Applied Research Laboratory. Bent beam specimens heat-treated to three strength levels are being exposed to a marine environment (Kure Beach, N. C.) and a semi-industrial environment (Monroeville, Pa.).

Chemical analysis and physical test results for these samples were presented in Report No. 4.

There has been no change in the status of the above test specimens over the past month. A duplicate of the cumulative test results is attached herewith as Table IV for reference purposes.

Similar bent beam specimens from the same heat of 4137 Co prepared by this project have been cut to length and mounted on an outdoor test rack on the roof of the Institute for heavy industrial environment exposure. As Table V shows, the specimens have been stressed to three percentage-of-yield-strength levels for each of the three tempers. While there were some failures to date, a comparison of results with Table IV shows that both the 550 and 750°F temper specimens exposed to the severe industrial environment at all "% of Y.S." levels appear to have outlasted (or nearly so) the 750°F temper specimens exposed to a milder or semi-industrial environment.

Stress corrosion testing of bent beam specimens from the Primary Heat of AM355 (cold-rolled to strength), secondary direction (direction of highest yield strength; 250, 260, and 300 Kpsi; in the rolling direction) is continuing. As Table VI shows, no failures have occurred to date for any of the strength levels under test.

TABLE IV

CUMULATIVE 4137 Co STRESS CORROSION BENT BEAM TESTS*

Primary (Lowest Y.S.) Direction

Austenitizing Temperature	Tempering Temperature	Approximate Tensile Stress**	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, days
Marine Exposure (Kure Beach, North Carolina)						
1700°F	550°F	190 kpsi	3	3	8	3 to 16
	750	160	5	5	13	6 to 18
	1100	130	5	None to date	--	--
		tests begun 10-26-60				
Semi-Industrial Exposure (Monroeville, Pennsylvania)						
1700°F	550°F	190 kpsi	5	5	19	2 to 30
	750	160	5	5	35	18 to 45
	1100	130	5	None to date	--	--
		tests begun 10-26-60				

* Cooperative testing program with U. S. Steel Applied Research Lab., Monroeville, Pa.
** All samples surface-stressed in holder to 75% of Y.S.

TABLE V

CUMULATIVE 4137 Co STRESS CORROSION BENT BEAM TESTS

Primary (Lowest Y.S.) Direction

Austenitizing Temperature	Tempering Temperature	% of Y.S. for Test	Approximate Tensile Stress	Number of Samples	Failures to Date	Average Time to Failures, days	Failure Time Range, days
Severe Industrial Exposure (Pittsburgh, Pennsylvania)							
1700°F	550°F	50	130 Kpsi	6	none to 59 days	--	--
	750	50	115	6	none to 59 days	--	--
	1100	50	95	6	none to 59 days	--	--
1700	550	75	195	6	none to 59 days	--	--
	750	75	175	6	none to 59 days	--	--
	1100	75	140	6	none to 59 days	--	--
1700	550	90	230	6	3 to 37 days	--	--
	750	90	200	6	3 to 59 days	--	--
	1100	90	170	6	none to 59 days	--	--

tests begun 3-2-61

TABLE VI

CUMULATIVE AM 355 STRESS CORROSION BENT BEAM TESTS

Secondary (Highest Y.S.) Direction*

Test Solution	Approx. Tensile Stress on Sample**	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, days
NaCl, 1 M	180 kpsi	6	none to 61 days	--	--
	195	6	none to 45 days	--	--
	226	6	none to 58 days	--	--
Na_2SO_4, 1 M	180	6	none to 60 days	--	--
	195	6	none to 45 days	--	--
	225	6	none to 58 days	--	--
$NaNO_3$, 1 M	180	6	none to 61 days	--	--
	195	6	none to 45 days	--	--
	225	6	none to 58 days	--	--
$NaPO_3$, 1 M	180	6	none to 60 days	--	--
	195	6	none to 45 days	--	--
	225	6	none to 55 days	--	--
Na_2S, 1 M	180	6	none to 60 days	--	--
	195	6	none to 45 days	--	--
	225	6	none to 58 days	--	--

* All samples stressed in holder to 75% of Y.S.

** The three Y.S. test levels of as-rolled material: 250 kpsi; 261 kpsi; and 302 kpsi.

U-Bend Tests

The cumulative U-bend stress corrosion test results for the low alloy, high strength 4137 Co specimens are given in Table VII. Samples of the highest strength level (550°F temper or a Y.S. of 250-260 Kpsi) are shown to be susceptible to all environments.

Table VIII shows the effect of water vapor exposure on identical samples of the same material (4137 Co). Of the three environments, dessicator (dry air), sealed container (saturated with water vapor), and laboratory environment (varying water vapor concentration), only the saturated air environment had an effect on the specimens.

The U-bend specimens of B120 VCA in the "as received" condition have had no failures to date, as indicated in Table IX.

U-bend specimens of the "as received" (cold rolled), low yield strength (196 Kpsi) PH15-7 Mo alloy have been immersed in the test solutions, namely, one molar NaCl, $NaNO_3$, Na_2S, Na_2SO_4 and $NaPO_3$. These specimens were taken in both the Primary (transverse to the rolling direction) and secondary (parallel to rolling direction) directions. The tensile specimens for PH15-7 Mo are now being machined and the physical properties will be presented in the next monthly report. Table X gives the cumulative data on the PH15-7 Mo U-bends and are designated as primary and secondary specimens.

TABLE VII

CUMULATIVE 4137 Co STRESS CORROSION U-BEND TESTS

Primary (Lowest Y.S.) Direction

Test Solution	Austenitizing Temperature	Tempering Temperature	Number of Samples	Failures to Date	Average Time to failure, days	Failure Time Range (days)
NaCl, 1M	1700°F.	550°F.	6	6	4.3	0.5 to 11.5
	"	750	6	2 to 283 days	-	--
	"	1100	6	none to 283 days	-	--
Na_2SO_4, 1M	"	550	6	6	0.9	10 min. to 2.5 days
	"	750	6	none to 223 days	-	--
	"	1100	6	none to 223 days	-	--
$NaNO_3$, 1M	"	550	6	6	1.2	0.5 to 1.5
	"	750	6	6	33.8	29.5 to 39.5
	"	1100	6	4 to 223 days	-	--
$NaPO_3$, 1M	"	550	6	6	4.5 min.	3 to 6 min.
	"	750	6	6	0.3 days*	34 min. to 5 days
	"	1100	6	6	1.5 days**	0.5 to 27 days
Na_2S, 1M	"	550	6	2 to 89 days	-	--
	"	750	6	1 to 89 days	-	--
	"	1100	6	none to 89 days	-	--

* One specimen lasting 5 days not averaged.
** One specimen lasting 27 days not averaged.

Note: Outer surface of U-bend samples is stressed beyond the Y.S.

TABLE VIII

CUMULATIVE 4137 Co STRESS CORROSION U-BEND TESTS

Primary (Lowest Y.S.) Direction

Environment	Austenitizing Temperature	Tempering Temperature	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, (days)
Dry Air (dessicator)	1700°F	550	3	none to 89 days	-	--
		750	3	none to 89 days	-	--
		1100	3	none to 89 days	-	--
Humid Air (satur. with water vapor)	1700°F	550	4	4	6.4	4 to 11.5
		750	4	3 to 81 days	-	--
		1100	4	none to 81 days	-	--
Laboratory (exposed directly to lab. environ.)	1700°F	550	4	none to 81 days	-	--
		750	4	none to 81 days	-	--
		1100	4	none to 81 days	-	--

Note: Outer surface of U-bend samples is stressed beyond the Y.S.

TABLE IX

CUMULATIVE B120 VCA STRESS CORROSION U-BEND TESTS

Secondary (Highest Y.S.) Direction

Test Solution	Y.S. (.2% Offset) Test Level, Kpsi	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, (days)
NaCl, 1M	135 (as recvd)	4	none to 115 days	-	--
$NaNO_3$, 1M	135 (as recvd)	4	none to 115 days	-	--
$NaPO_3$, 1M	135 (as recvd)	4	none to 115 days	-	--

Note: Outer surface of U-bend samples is stressed beyond the Y.S.

TABLE X

CUMULATIVE PH15-7 Mo STRESS CORROSION U-BEND TESTS

Test Solution	Direction of Specimen	Number of Specimens	Failures to Date	Average Time to Failure, days	Failure Time Range, days
NaCl	Primary	6	none to 32 days	--	--
	Secondary	6	none to 32 days	--	--
$NaNO_3$	Primary	5	none to 32 days	--	--
	Secondary	6	none to 32 days	--	--
Na_2S	Primary	6	none to 32 days	--	--
	Secondary	6	none to 32 days	--	--
Na_2SO_4	Primary	5	none to 32 days	--	--
	Secondary	6	none to 32 days	--	--
$NaPO_3$	Primary	5	none to 32 days	--	--
	Secondary	6	none to 32 days	--	--

Note: Outer surface of U-bend specimens stressed beyond the Y.S. Primary (lowest Y.S.) and Secondary (highest Y.S.).

U-bend specimens of AM355 (Primary direction, 226 Kpsi Y.S.) have been immersed in the test solutions. As can be seen from Table XI, the specimens show a susceptibility to stress corrosion in the one molar NaCl solution. The above observation is based on only 6 specimens; however, additional specimens are being prepared to confirm the observation.

In a previous section of this report, sample preparation of Ardeform 301 was presented in detail. Some data has been obtained on these specimens, and, again, the samples indicate susceptibility to stress corrosion in the one molar NaCl solution, as was the case for the AM355 U-bends. Cumulative Ardeform 301 stress corrosion data is shown in Table XII.

TABLE XI

CUMULATIVE AM355 STRESS CORROSION U-BEND TESTS

Primary (Lowest Y.S.) Direction

Test Solution	Y.S. (.2% Offset) Test Level Kpsi	Number of Specimens	Failures to Date	Average Time to Failure, days	Failure Time Range, days
NaCl	226 (as received)	6	6	3.25	0.5 to 8
$NaNO_3$	226 (as received)	6	none to 26 days	--	--
Na_2S	226 (as received)	6	none to 24 days	--	--
Na_2SO_4	226 (as received)	6	none to 26 days	--	--
$NaPO_3$	226 (as received)	6	none to 26 days	--	--

Note: Outer surface of U-bend specimens stressed beyond the Y.S.

TABLE XII

CUMULATIVE ARDEFORM 301 STRESS CORROSION U-BEND TESTS

Stress Corrosion Environment	Sample Direction	Test Surface in Tension	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, days
Sample No. 1						
NaCl, 1M	Longitudinal	Outside (convex)	4	4	1.25	0.5 to 2
	Longitudinal	Inside (concave)	4	2 to 26 days	--	--
	Trans.	Outside	6	none to 26 days	--	--
$NaNO_3$, 1M	Long.	Outside	4	none to 26 days	--	--
	Long.	Inside	4	none to 26 days	--	--
	Trans.	Outside	6	none to 26 days	--	--
Na_2S, 1M	Long.	Outside	4	none to 26 days	--	--
	Long.	Inside	4	none to 26 days	--	--
	Trans.	Outside	6	none to 26 days	--	--
Na_2SO_4, 1M	Long.	Outside	4	none to 26 days	--	--
	Long.	Inside	4	none to 26 days	--	--
	Trans.	Outside	6	none to 26 days	--	--
$NaPO_3$, 1M	Long.	Outside	4	none to 24 days	--	--
	Long.	Inside	4	none to 24 days	--	--
	Trans.	Outside	6	none to 24 days	--	--

Note: Outer surface of U-bend samples is stressed beyond Y.S.

IV. FUTURE WORK

Sample preparation, heat treatment surveys, heat treatment, metallographic examination, and stress corrosion testing of the assigned alloys and comparative heats will constitute the research effort for the next few months.

Continuing cumulative bent beam and U-bend stress corrosion tests results will be presented as they become available.

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